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Use of sputtered zinc oxide film on aluminium foil substrate to produce a flexible and low profile ultrasonic transducer

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ABSTRACT

A flexible and low profile ultrasonic transducer was fabricated for non-destructive testing (NDT) applications by DC sputtering of 3 μm thick, c-axis oriented, ZnO film on 50 μm aluminium foil. Due to the thin foil-based construction, the transducer can be applied to curved objects and used in sites of restricted accessibility. The device has been used to demonstrate detection of simulated defects in a 45 mm diameter steel pipe, and for thickness measurement on a 3.1 mm thick flat carbon steel plate. Centre frequency measured on the flat plate was 24-29 MHz, with -6dB bandwidth 4-7 MHz. The pulse duration depended on the couplant, at best 3 cycles or 0.12 μs using SONO

Ultragel or epoxy couplant. Transducer performance was found to be comparable to a commercial 10 MHz piezoelectric ultrasonic transducer.

Keywords: zinc oxide, thin film, DC sputtering, flexible ultrasonic transducer, non-destructive testing.

1. Introduction

Ultrasonic non-destructive testing (NDT) is widely used in industry for defect detection, material property studies, process analysis, and structural health monitoring. However, the surfaces to be inspected may have rather complex geometries, and sometimes extremely limited site accessibility. In these cases, conventional planar ultrasonic transducers, typically made of monolithic piezoelectric ceramics, may be too bulky and non-conforming to enable a thorough inspection.

To solve this problem, a number of different approaches have been taken to developing flexible and low profile ultrasonic transducers. Use of polyvinylidene fluoride (PVDF) piezoelectric polymer films has been demonstrated [1], although the low acoustic impedance (2.7 MRayl), together with a low electromechanical coupling coefficient and low Curie temperature, mean that PVDF can have only limited use in NDT applications.

Casula et al. [2,3], designed a “smart phased array transducer” composed of 24 rigid piezoelectric elements, mechanically assembled with helical springs to form a conformable linear array of 48×20 mm^2 dimensions, and incorporating a profilometer to provide information to calculate modified focal laws for operation on an irregular non-planar surface. The device could conform to surfaces with local concave or convex curvatures down to 15 mm in radius for use on cooling pipes in a pressurized water reactor, where conventional wedge transducers had been unable to operate satisfactorily. However this transducer is not low profile.

Bowen et al.[4] fabricated a broad-band and low profile flexible piezoelectric transducer using commercially available 800 μm diameter cylindrical PZT-5A fibres, lapped flat, electroded, alligned parallel with 0.8 mm gap between two neighbouring fibres, and sandwiched between two copper-coated Kapton sheets. The transducer was conformable in one direction, with the axis of curvature along the fibre direction.The resonance frequency of the completed transducer was just below 2 MHz. A 10 mm diameter hole along the central axis of a 38 mm diameter steel rod was detected, with the transducer applied using commercial couplant gel.

Flexible piezoelectric-polymer composites, including 0-3 [5], 2-2 [6], but mostly 1-3 connectivity [7-9], have also been investigated. It has proved difficult to find a compromise between maintaining a suitable piezoelectric pillar aspect ratio in the piezocomposite to ensure that the device has a well-defined thickness mode performance, and minimizing the ceramic volume fraction to ensure conformability. Embedding piezoelectric tiles known as ‘platelets’ in a polymer matrix was shown to offer some increased flexibility over conventional 1-3 configurations while demonstrating reasonably good uni-modal performance in the 4 to 7 MHz frequency range [8,9]. Harvey et al. [7] incorporated a random arrangement of 250 µm diameter PZT 5A fibres (oriented perpendicular to the plane of the device) into the platelets and constructed a so-called random fibre Composite Element Composite Array Transducer (CECAT). The random microstructure removed parasitic inter-pillar modes to give comparable performance to a conventional 1-3 piezocomposite whilst maintaining conformability.

Kobayashi et al. [10-13], and most recently Wu et al. [14,15], developed a low-profile transducer consisting of a piezoelectric ceramic film (PZT, Bismuth Titanate (BIT), or PZT/BIT mixture) of 40-120 µm thickness, fabricated using sol-gel sprays on a stainless steel or titanium metal foil 30-75 µm thick acting as the front electrode. The back electrode was silver paste. The porosity (>20% volume) of the ceramic film after firing, together with the use of the thin metal foil, allowed the transducer to be bent to a curvature of 15 mm radius, whilst operating up to 160 ºC at 10 MHz with a signal to noise ratio (SNR) of 36 dB [11].

Our design for a low profile and flexible ultrasonic transducer consists of a thin film piezoelectric (ZnO) layer on an Al foil substrate. The thin film was fabricated by DC magnetron sputtering, a physical vapour deposition technique fully compatible with electronic integrated circuit (IC) technologies. Transducers made this way can be low profile (<100 µm) and possess excellent flexibility to conform to a curved surface. Sputtered ZnO films normally grow preferentially along the c-axis or (002) orientation, which is the required piezoelectric orientation, and no poling is required. ZnO films have previously been deposited on Al foils of varying thicknesses to develop Lamb wave devices [16], strain sensors [17], and very high frequency concace focused transducers [18,19] for acoustic microscopes and underwater imaging applications.

A thin film of ZnO 3-5 μm thick has a fundamental $\lambda/2$ thickness mode resonance frequency of 600 MHz to 1.5 GHz, whereas ultrasonic inspections are usually carried out in the frequency range of a 500 kHz to 30 MHz. However our previous work on thin film transducers has shown that they are able to operate far below their thickness mode resonance frequency. In fact the operating frequency of the transducer is determined primarily by the bandwidth of the excitation pulse and receiver system [20, 21]. In this way, sputtered thin film transducers directly deposited onto metal components were shown to be capable of use in NDT applications [22-24]. Using aluminium nitride (AlN) films with, V_L = 10 000 m s^{-1}, 3-6 μm thick, and 2-4 mm transducer diameter, directly deposited onto solid metal substrates, we found a 10-40 MHz operating frequency range when driven by a JSR Ultrasonics DPR300 Pulser/Receiver (bandwidth 50 MHz) reducing to 1-10 MHz when driven by a USK7 flaw detector (bandwidth 10 MHz). The 10 MHz signals generated by a 6 μm thick AlN film transducer penetrated a total distance of 260 mm in ferritic steel. Typical axial resolution at 25 MHz was 3-4 cycles, duration 0.12-0.16 μs. Here we use ZnO thin films on a flexible foil substrate, which gives the possibility for greater utility in ultrasonic NDT inspections. Performance of the flexible transducer on both flat and curved surfaces was examined through pulse-echo experiments and simulated defects were detected.

2. Transducer fabrication and characterisation

A 50 µm thick commercially available Al foil was used as the substrate for the flexible transducers. A ZnO film of 3 µm nominal thickness was deposited by DC magnetron sputtering using a 100×300 mm^2 Zn target. Detailed sputtering conditions were as follows: plasma power 400 W, chamber pressure 3.5 mTorr, Ar/O_2 flow ratio 50/50 sccm, and target to substrate distance 20 mm. The typical deposition rate was about 300 nm/hr, or 0.3 µm/hr. No intentional substrate heating was implemented, and the temperature stayed below 80 °C. Throughout the deposition process, the drum-type substrate holder was kept in constant rotation at 50 rpm in around the target to improve film uniformity over a 50 x 70 mm^2 deposition area. As shown in Figure 1, the Al foil remained wrinkle free after the deposition, suggesting low internal stress. Foil ductility and flexibility was virtually unaffected.

Figure 2 shows a cross-sectional scanning electron microscopy (SEM) image of the ZnO film on the aluminium foil substrate. The film has formed a dense polycrystalline columnar microstructure, with the growth direction aligned normal to the substrate surface. Figure 3 shows an XRD analysis which confirms c-axis (002) growth.

The ZnO film was further characterized using an HP 8752A Network Analyser. For this investigation the aluminium foil substrate was used as the bottom electrode and sputtered Cr/Au 20/100 nm thick was used as the top electrode. Figure 4 shows the return loss S_{11} of the device, confirming the thin film resonance frequency (local minimum of S_{11}) of 1.02 GHz, corresponding to the $\lambda/2$ thickness mode resonance of a 3 µm film, based on accepted values for V_L in ZnO of 6000-6400 m s^{-1}.

To produce ZnO thin film transducers for the ultrasonic NDT experiments, the ZnO/Al foil was cut into rectangular strips, and unless otherwise specified sodium silicate based adhesive with a solid content of 42% (High Temperature L-7 Binder, Heatflo Sealants Ltd.) was used to bond the transducers, foil side down, to the component being inspected. The foil substrate acted as the front electrode of the transducers. Silver-loaded paint, (Electrodag 1415, Agar Scientific Ltd), was applied in 2-4 mm diameter drops to the top surface of the film to form the back electrode and define the size of each transducer. The bonding adhesives used in the current investigation were mostly nonconductive, so the metal component could not be used as the ground, therefore the ZnO film was scraped off at one end of each rectangular strip to expose the aluminium foil surface to allow electrical connections. Figure 5 shows (a) a schematic diagram illustrating how the flexible transducers are deployed on a component, and (b, c) images of the flexible transducer bonded to a carbon steel plate and a skimmed carbon steel pipe.

3. Transducer performance

Ultrasonic performance of the transducers was examined by pulse-echo measurements using a JSR Ultrasonics DPR300 Pulser/Receiver (bandwidth 50 MHz). The frequency response of the transducer was obtained using a 512-point Fast Fourier Transform (FFT) applied to the return echoes. Signals were multiplied by a Hanning window to reduce the spectral leakage, with two thirds of each FFT section overlapped.

Figure 6 shows the performance on a 3.1 mm thick carbon steel plate of the flexible ZnO thin film transducer compared with that of a 10 MHz commercial transducer (Sonatest Img5010). The flexible ZnO transducer was bonded onto the plate by applying a thin layer of sodium silicate adhesive to both the transducer and plate, which were then immediately sandwiched between two PTFE plates and firmly clamped in a vice for at least 24 hours to allow the adhesive to set. The 10 MHz commercial transducer was used with a liquid couplant (Sono Ultragel), pressed by hand.

The first four echoes from each transducer are displayed in the A-scans, corresponding to a total ultrasonic propagation distance of 24 mm. Frequency spectra were obtained for the 1st echo in each case, showing that the nominally 10 MHz commercial transducer was actually operating at 4.3 MHz with a -6 dB bandwidth of 0.8 MHz, and the flexible transducer was operating at 25.8 MHz with a -6 dB bandwidth of 4.6 MHz. It can be noted that there was no observable attenuation from the 1st to the 4th echo for the 4.3 MHz signal from the 10 MHz commercial transducer, whereas the 25.8 MHz signal from the flexible transducer was attenuated by 12 dB. The signal from the flexible transducer typically consisted of 4-5 cycles (pulse duration 0.18 μs). The commercial transducer, on the other hand, showed more ringing, the echo typically lasting 6-9 cycles (pulse duration 0.6 μs). This implies that the flexible transducer would have better depth resolution in ultrasonic inspections.

Figure 7 shows the performance of the flexible transducer on a 3.1 mm thick carbon steel plate when different acoustic coupling/bonding methods were employed (see Table 1). The sodium silicate and epoxy adhesives showed large signal to noise ratio, and short pulse durations. Use of cyanoacrylate superglue produced the least signal attenuation but longest pulse duration. Use of the liquid couplant, on the other hand, produced a low SNR of 4, however it must be noted that in this case there was no additional pressing or clamping mechanism involved. The flexible transducer was only ‘attached’ to the steel plate by the aqueous couplant gel.

Figure 8 illustrates the transducer performance on a 44.7/ 28.9 mm O.D./I.D. carbon steel pipe. The flexible transducer was attached to the outer surface of the pipe using sodium silicate adhesive. The curvature in this case was 0.05. To make sure that a thin and uniform bonding layer was formed, the transducer was firmly bound to the pipe under several rounds of PVC insulation wrap for at least 24 hours to allow the glue to set. The flexible transducer demonstrated very good performance on the curved surface. Figure 8(a) shows the first three back wall echoes. The average time between two consecutive echo peaks was 2.63 μs, corresponding to the measured wall thickness of the pipe 7.9 mm with speed of sound 6000 m s^{-1}. In this case the centre frequency was 13 MHz with a -6dB bandwidth of 8 MHz. Note that the noise floor in Figure 8 was depressed because the concave transducer created a focusing effect.

In order to investigate detection of a simulated defect, a slot was hand-sawed into the inner surface of the pipe along the axial direction. The slot was 2 mm wide with a tapering depth profile along the length direction. The flexible transducer was bonded onto the outer surface of the pipe directly above the slot. An array of four equally spaced back electrodes, each 3 mm diameter, were applied on the top of the ZnO film. Figure 9 shows (a) an image of the slot, and (b) a schematic diagram of the relative position of each individual back-electroded transducer (BET1 to BET4) along the slot length, and slot depth at the centre of each transducer.

Figure 10 shows the pulse-echo results from each transducer, including a backwall echo shown as a reference. We can see that the flexible transducer has good sensitivity in mapping the tapering slot profile. Time delays of 0.79, 0.62, 0.54 and 0.42 μs were recorded from transducers BET1 to BET4 between the backwall echo and the signal from the saw cut. The slot depth underneath each transducer was therefore estimated as 2.4, 1.9, 1.6, and 1.3 mm (taking the speed of sound to be 6000 m s^{-1}), giving close agreement to the actual measured slot depths of 2.5, 1.9. 1.4, 1.3 mm at the transducer positions.

4. Conclusions

This paper presents an innovative design of flexible low profile transducers, made from sputtered thin films of ZnO. Transducers made of 3 μm ZnO sputtered on a 50 μm aluminium foil substrate demonstrate excellent conformability and are therefore particularly suitable for ultrasonic inspections of irregular geometry and limited accessibility.

The flexible transducers operated in a frequency range of 15 to 30 MHz with pulse duration around 0.15 μs. They showed good performance on both flat and curved surfaces, and satisfactory sensitivity in defect detection. The transducers worked best when bonded with epoxy or sodium silicate based adhesives.

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Table Caption:

Table 1: Performance of the ZnO thin film on Al foil flexible transducer in pulse echo operation on a 3.1 mm thick flat carbon steel plate with different bonding/coupling methods.

Figure Captions:

Figure 1: Sputtered ZnO on Al foil substrate used to make flexible low profile transducers. The ZnO/Al foil can be easily cut using scissors.

Figure 2: SEM cross-section of the sputtered ZnO thin film, grown in (002) orientation, showing a dense columnar microstructure.

Figure 3: XRD analysis of 3 μm sputtered ZnO film on 50 μm Al foil.

Figure 4: Return loss (S_{11}) for 3 μm sputtered ZnO film on 50 μm Al foil, measured using network analyzer. Resonance frequency shown by local minimum at 1.02 GHz.

Figure 5: (a) Schematic diagram of flexible transducer. Transducers bonded to (b) flat carbon steel plate 3.1 mm thick; (c) carbon steel pipe 45 mm diameter. Circular drops of silver paint formed the back electrodes.

Figure 6: Performance comparison on a 3.1 mm thick flat carbon steel plate of commercial 10 MHz transducer using Sono Ultragel liquid couplant (a, b); sputtered ZnO flexible transducer using sodium silicate based adhesive as couplant (c, d). Left: pulse-echo results; right: frequency components (main graph) and first echo (inset, with μs scale).

Figure 7: Performance of flexible transducers on a 3.1 mm thick flat carbon steel plate using different adhesive bond layers or couplant: (a, b) epoxy; (c ,d) superglue; and (e, f) liquid couplant (Sono Ultragel). Left: pulse-echo results; right: frequency components (main graph) and first echo (inset, with μs scale).

Figure 8: Performance of the flexible transducer using sodium silicate based adhesive as couplant on a 44.7 mm O.D. carbon steel pipe section with a wall thickness of 7.9 mm, (a) pulse-echo results; (b) frequency components of the 1st echo (inset).

Figure 9: (a) image showing the tapering depth profile of the slot. (b) schematic diagram showing the relative location of each back-electroded transducer (BET1-4) along the slot length direction (dimensions in mm, not to scale).

Figure 10: Pulse-echo results collected from (a) pipe backwall; (b) BET1; (c) BET2; (d) BET3; (e) BET4. Time delays between the backwall echo and echoes from each back-electroded transducer (BET1 to BET4) were marked to obtain the slot depth profile.

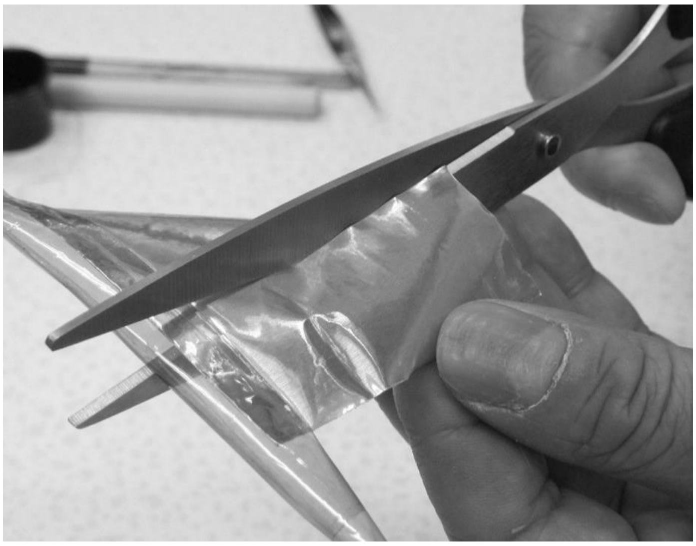


Figure 1

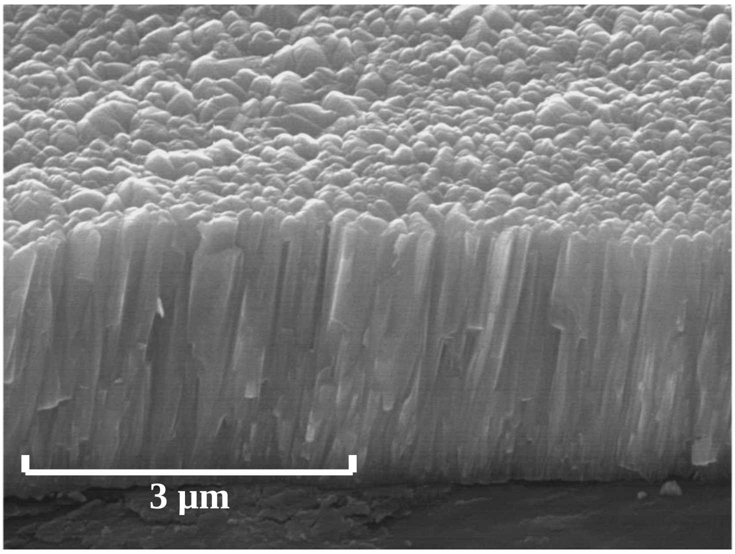


Figure 2

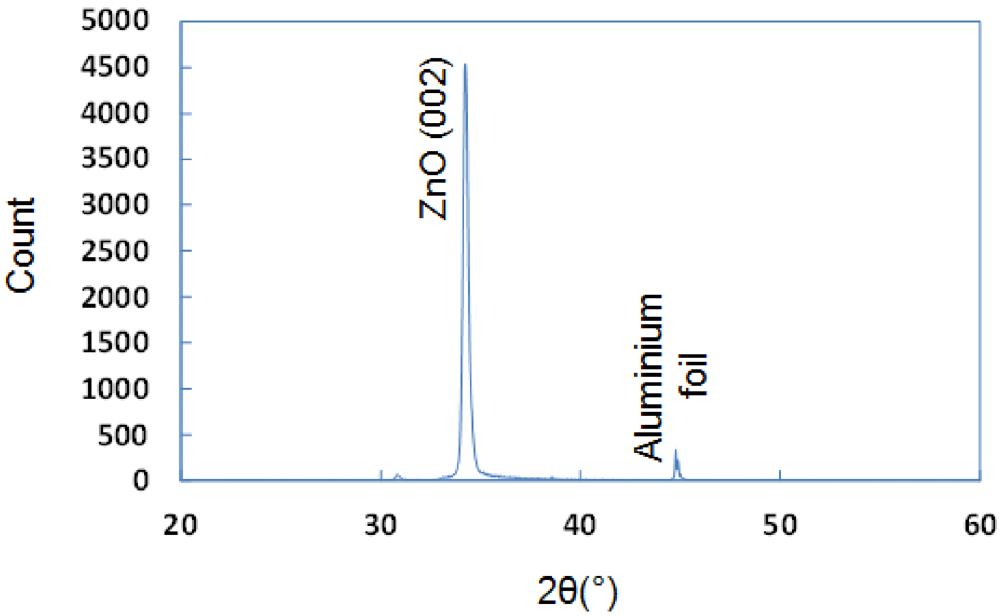

5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
Count
ZnO (002)
Aluminium foil
20
30
40
50
60
2θ(°)

Figure 3

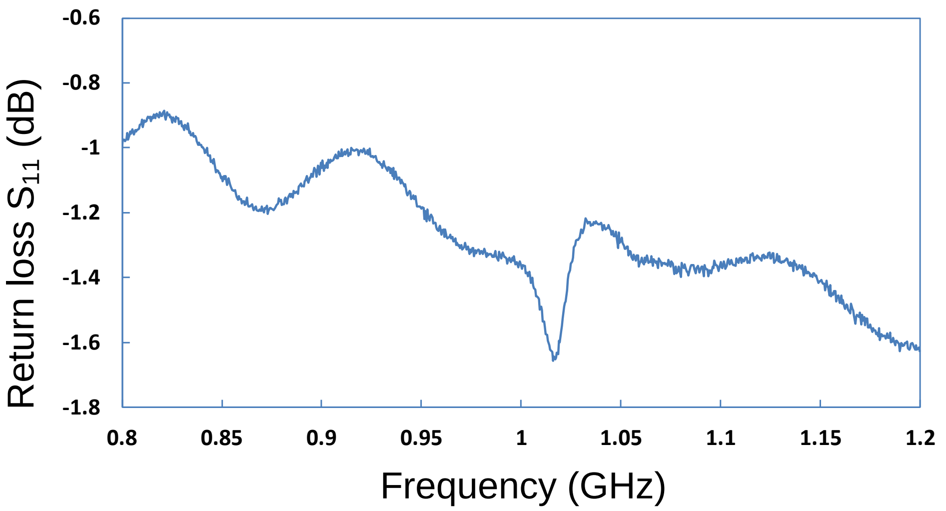

-0.6
-0.8
-1
-1.2
-1.4
-1.6
-1.8
Return loss S11 (dB)
0.8
0.85
0.9
0.95
1
1.05
1.1
1.15
1.2
Frequency (GHz)

Figure 4

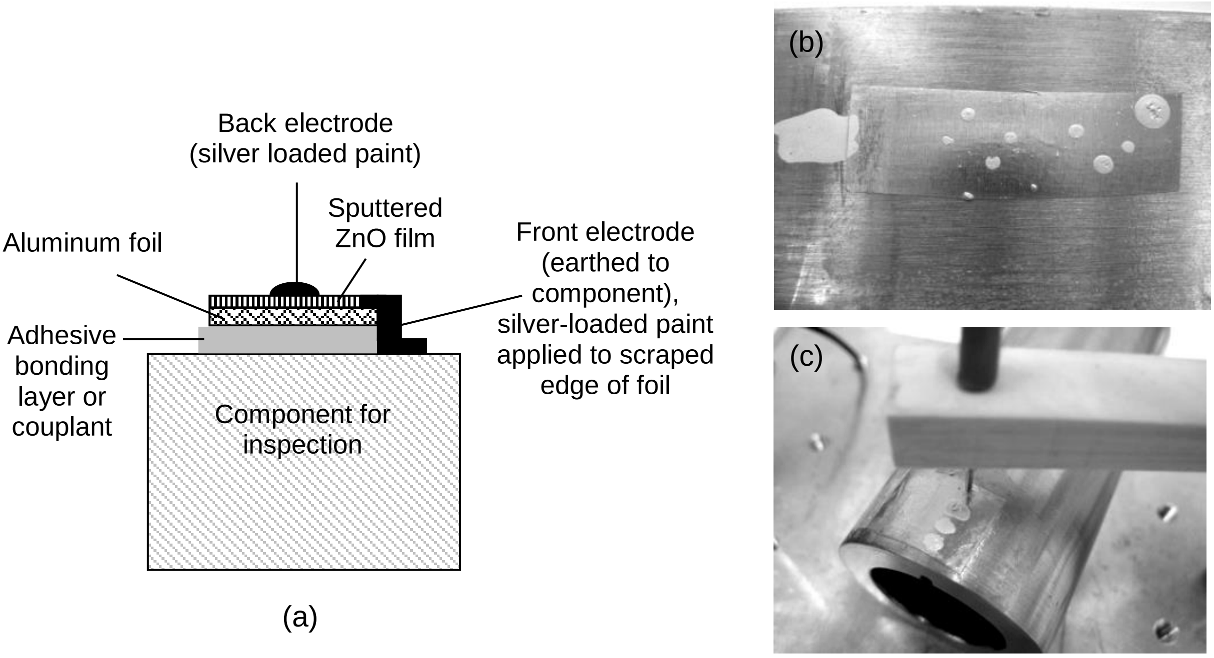

Back electrode
(silver loaded paint)
Sputtered
ZnO film
Aluminum foil
Front electrode
(earthed to
component),
silver-loaded paint
applied to scraped
edge of foil
Adhesive
bonding
layer or
couplant
Component for
inspection
(a)
(b)
(c)

Figure 5

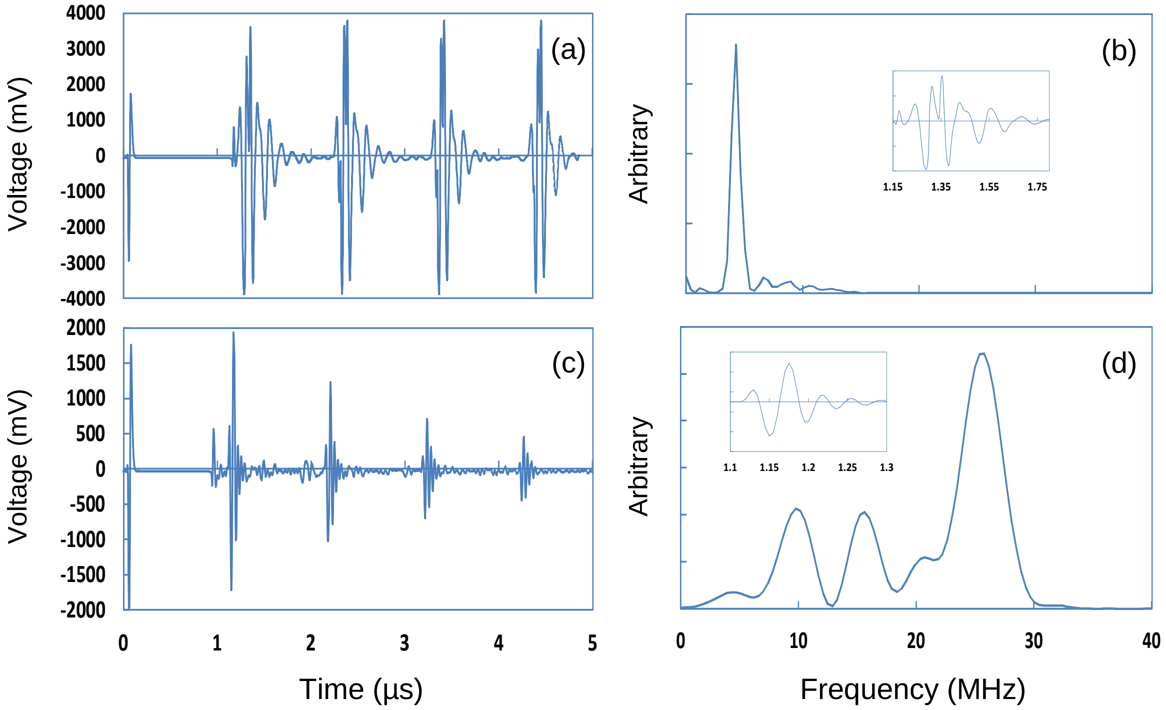

Voltage (mV)
4000
3000
2000
1000
0
-1000
-2000
-3000
-4000
(a)
Arbitrary
(b)
1.15
1.35
1.55
1.75
Voltage (mV)
2000
1500
1000
500
0
-500
-1000
-1500
-2000
(c)
Arbitrary
(d)
1.1
1.15
1.2
1.25
1.3
0
1
2
3
4
5
Time (µs)
0
10
20
30
40
Frequency (MHz)

Figure 6

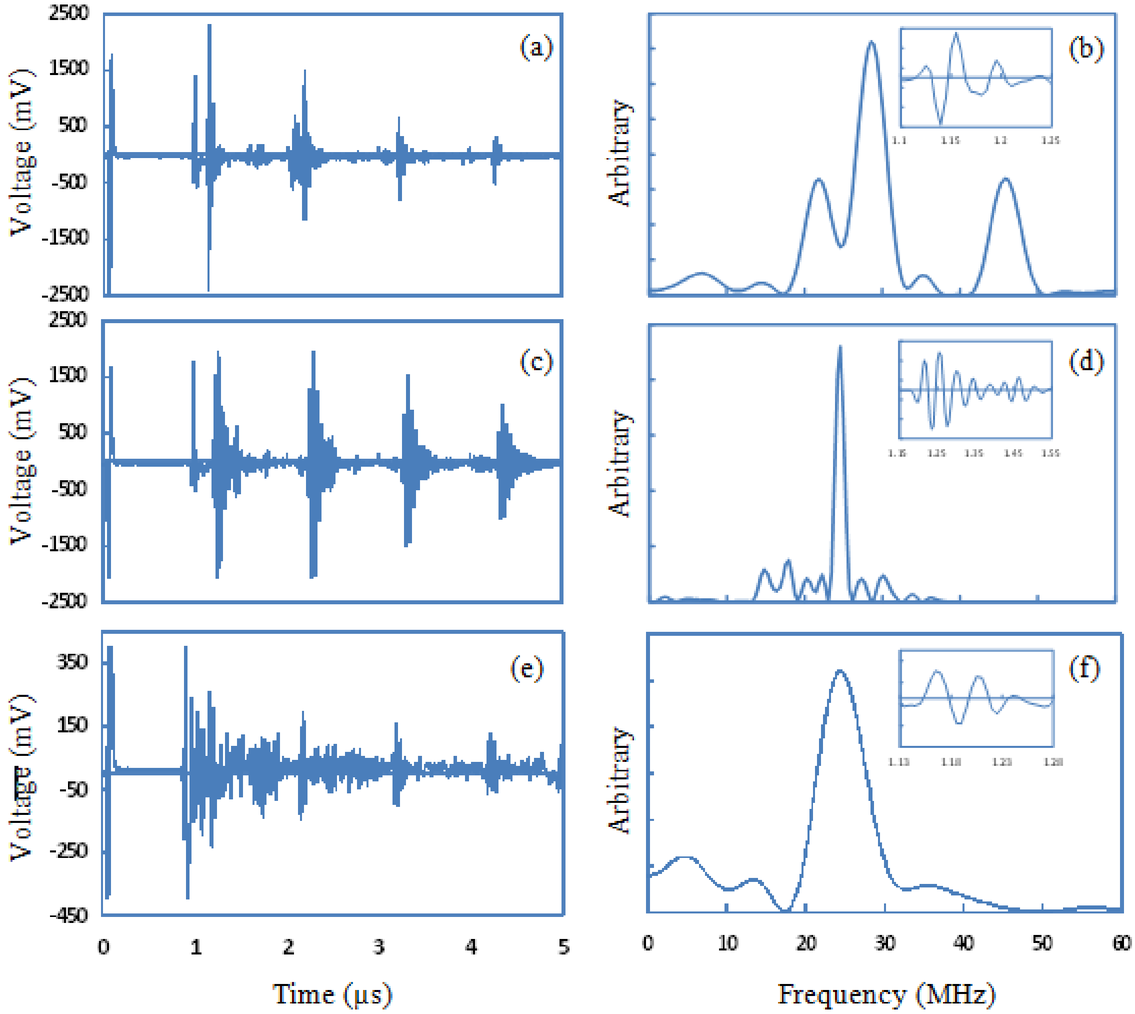

2500
1500
500
-500
-1500
-2500
Voltage (mV)
(a)
Arbitrary
(b)
(c)
(d)
350
150
-50
-250
-450
(e)
(f)
0
1
2
3
4
5
Time (μs)
0
10
20
30
40
50
60
Frequency (MHz)

Figure 7

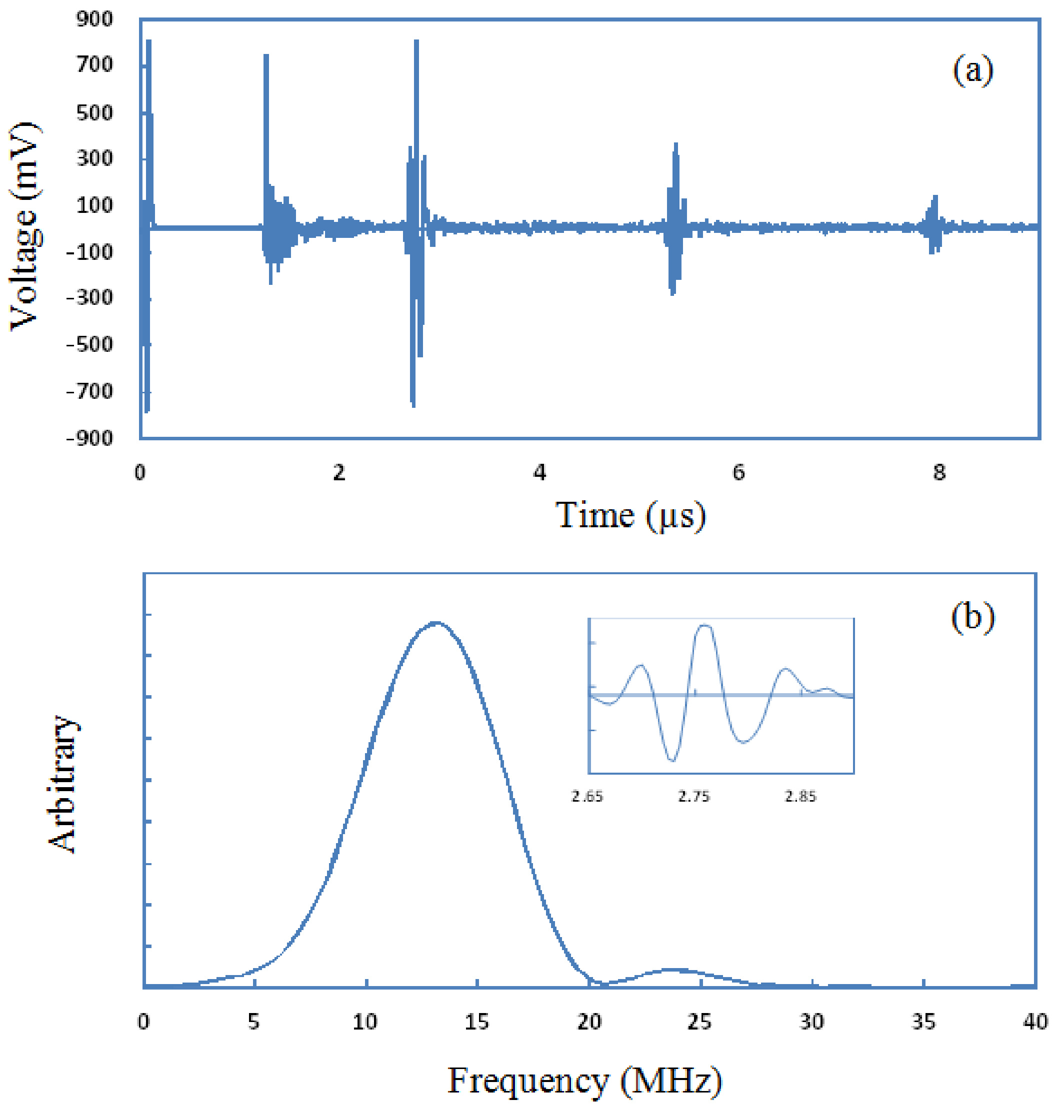
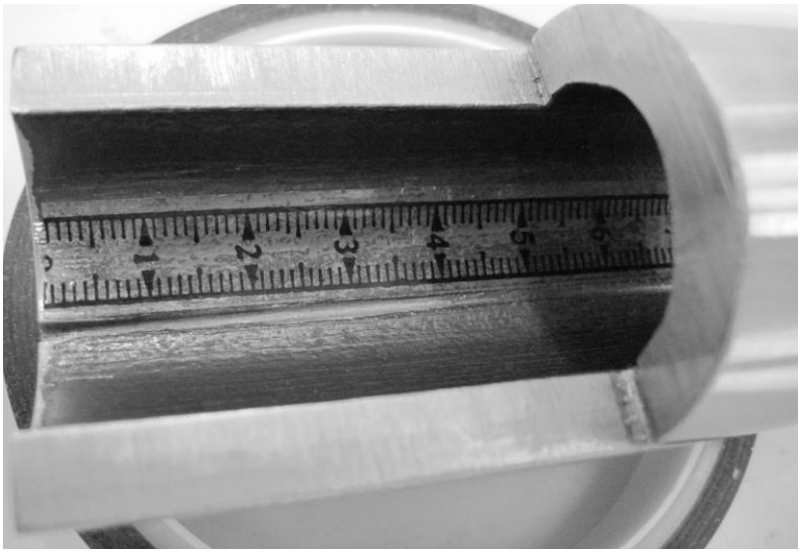
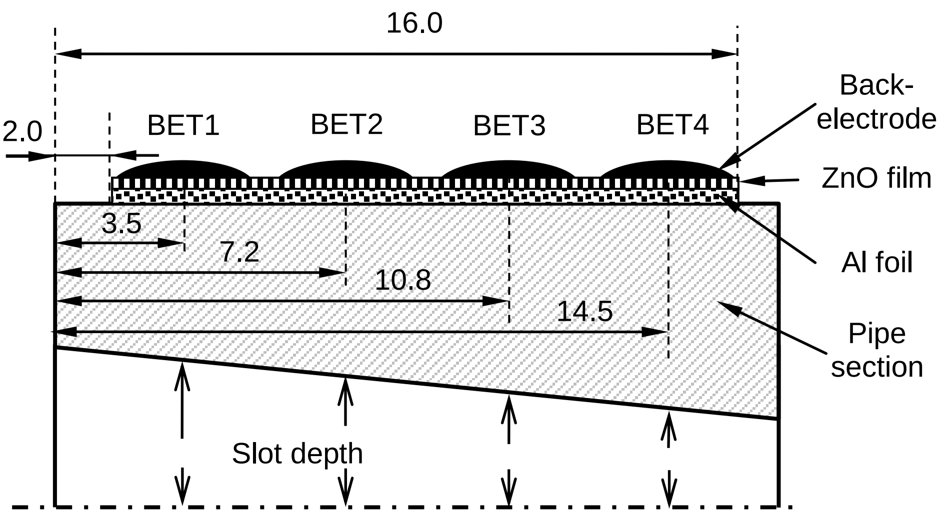

900
700
500
300
100
-100
-300
-500
-700
-900
Voltage (mV)
(a)
0
2
4
6
8
Time (μs)
(b)
Arbitrary
2.65
2.75
2.85
0
5
10
15
20
25
30
35
40
Frequency (MHz)

Figure 8



(a)


16.0
2.0
BET1
BET2
BET3
BET4
Back-
electrode
ZnO film
Al foil
3.5
7.2
10.8
14.5
Pipe
section
Slot depth

(b)

Figure 9

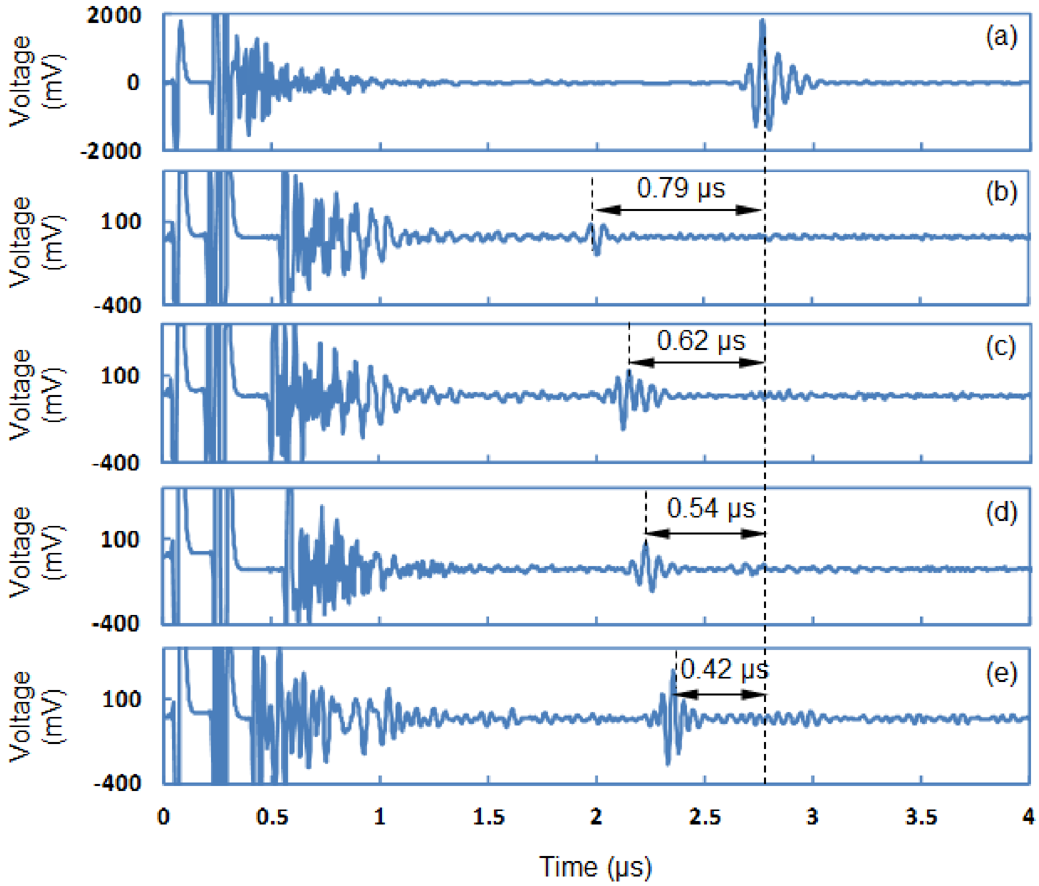

Voltage (mV)
2000
0
-2000
(a)
100
-400
0.79 μs
(b)
0.62 μs
(c)
0.54 μs
(d)
0.42 μs
(e)
0
0.5
1
1.5
2
2.5
3
3.5
4
Time (μs)

Figure 10

Couplant or bonding medium	**First Echo**				**Signal attenuation from 1st to 4th echo (dB)**
	Pulse duration	**Signal to noise ratio**	**Centre frequency (MHz)**	**-6dB bandwidth (MHz)**	
Sodium silicate adhesive	4 cycles or 0.17 µs	25	26	4.6	11
Epoxy	3 cycles or 0.12 µs	29	29	4.4	16
Cyanoacrylate superglue	7 cycles or 0.29 µs	22	25	7	6
SONO Ultragel couplant	3 cycles or 0.13 µs	4	24	7	9

Table 1